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SEMICONDUCTOR PROPERTIES NEAR INTERFACES

Final Technical Report

Grant No. AFOSR 77-3419

Aug. 1, 1977 - July 31, 1980

D. B. Wittry

S. Y. Yin

F. Guo

University of Southern California
Los Angeles, CA 90007

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I Abstract

Surface layers on GaAs were studied using cathodoluminescence and secondary ion mass spectrometry. The layers studied included thermally-converted layers formed on semi-insulating GaAs using various heat treatments, and oxides grown by anodizing in aqueous and non-aqueous electrolytes. Some of the investigations required improvements in the Ion Microprobe Mass Analyzer; in the course of the investigations an improved ion detector was developed and a microcomputer-based automation system was constructed.

II. Experimental Work and Results

1. Thermal conversion of semi-insulating GaAs

The principal subject of the present investigations was the study of thermal conversion of Cr-doped semi-insulating GaAs when heat treated in flowing hydrogen and when heat treated by the melt-controlled ambient technique. By using specimens subjected to different heat treatments and by using results of both photoluminescence and cathodoluminescence, it has been possible to propose a model for the conversion of the surface of semi-insulating GaAs to p type. Cathodoluminescence spectra at 30 degrees K show emission bands at 1.496 eV, 1.407 eV, 1.363 eV and 1.23 eV. These bands are attributed to acceptor levels introduced through the site transfer of silicon and carbon atoms, or to copper contamination during the annealing process. An emission band at 1.363 eV is attributed to transitions involving levels due to Si donors and Si acceptor Ga vacancy complex. The latter emission band is not seen in low temperature photoluminescence spectra. It is believed that this is due to the difference in sampling depth of the photoluminescence and cathodoluminescence techniques.

In photoluminescence, the sampling depth is of the order of 1/4 micron due to the strong absorption of the light. In cathodoluminescence at 40 keV, the mean excitation depth is about 2 microns which is approximately the same as the thickness of the thermally converted layer. The diffusion coefficient for Ga in GaAs is much greater than the diffusion coefficient for As. Moreover, it is well known that in the thermal decomposition of GaAs, there is a greater loss of As than of Ga. Thus it is expected that there will be a high concentration of As vacancies near the surface and that the Ga vacancies will have a lower concentration but extending to larger distances from the surface.

The 1.363 eV transition may be due either to copper on a Ga site or to a complex involving an Si acceptor - Ga vacancy complex. This ambiguity, as well as the relative contributions of Si and C as impurities, should be resolvable by depth profiles using secondary ion mass spectrometry. However, it was not possible to get good data from our IMMA instrument operated in the manual mode over long periods of time as are required to obtain depth profiles of these layers. These investigations will be possible using another SIMS instrument recently placed in operation in our laboratory and will be continuing under a new AFOSR grant. The results of the work so far are described in a manuscript included in the appendix.

2. Study of anodic oxides on GaAs

The second area of experimental work has been the study of anodic oxides on GaAs using cathodoluminescence and secondary ion mass spectrometry (SIMS). To some extent, this work was exploratory in nature since there has been no prior work on the cathodoluminescence of these oxides. The results of room temperature cathodoluminescence show some promise for this technique - namely, the presence of Ga_2O_3 was clearly indicated, oxide layers down to 100 Å in thickness could be detected, and changes in spectra after annealing in hydrogen at 440 degrees C were observed but no changes were seen after annealing in nitrogen. However, the weak intensities of peaks that might be associated with As_2O_3 or As_2O_5 indicate that considerable further work needs to be done before a clear interpretation of the cathodoluminescence results can be made.

Some of the specimens studied in this work had been previously studied by SIMS using a quadrupole mass spectrometer instrument (the QMAS) at Applied Research Laboratories. The SIMS results confirmed the changes that were seen in the cathodoluminescence spectra after annealing in hydrogen. SIMS results obtained at Applied Research Laboratories also indicated the presence of sodium in some of the anodic oxides and a piling up of sodium at the oxide-semiconductor. Whether this was an intrinsic property of the samples or an artifact of the measurement is not known. The data were obtained with a positive primary beam and it is possible that the surface charging of the oxide caused the drift of sodium ions toward the interface. While one attempt was made to repeat the results with a negative ion beam, the results were inconclusive and shortly thereafter the instrument was no longer available due to the principal operator leaving ARL.

SIMS results were also obtained on the impurities present in the oxide layers using the IMMA at USC. These results also showed the presence of high sodium concentration. SIMS spectra from oxides grown in ammonium pentaborate showed the presence of boron.

A number of depth profiles were made in the anodic oxides using the IMMA. However, because of the large number of experimental parameters and the difficulty of controlling all of these parameters when operating in a manual mode the results are questionable (since the termination of the grant, the computer automation system described in the following section was put into operation).

The results obtained so far on the study of anodic oxides on GaAs are described in a manuscript included in the appendix.

III. Advances in Instrumentation for SIMS

1. Development of an improved ion detector

In the course of the experimental work, it was necessary to construct a new detector for the IMMA instrument. This was due to limitations of the Daly-type detector which was provided on our prototype IMMA instrument; this detector did not provide good separation of signal and noise as required for minimum detection limits for impurities. The detector which was designed to take maximum advantage of existing components is a hybrid detector combining several stages of electron multiplication with a scintillation counter. This detector, described in the appendix, provides very low background without sacrifice of detection efficiency.

2. A microprocessor-based computer system for control and data recording with SIMS instruments

As indicated in the foregoing, it has been very difficult to obtain reliable depth profile data with the IMMA instrument when operated in a manual mode. For this reason, a computer-controlled system was designed and constructed. The principal novel feature of this system is the use of an analog multiplier-divider to provide for coarse and fine mass settings so that equal fine mass setting can be used to step over a given mass peak with instruments using a magnetic spectrometer. Thus, the programming can be simplified and a relatively small computer can be used. This approach was described in a paper presented at the second international conference on SIMS and has been implemented using a PET microcomputer with 16 K bytes of memory (PET model 2016).

During the summer of 1980 students working in our laboratory developed software and additional hardware to control the IMMA instrument from the PET microcomputer. This system provides for the selection of any number of masses, stepping over each mass peak, recording and display of all data recorded in the last spectrum scanned, selection of the number of scans, recording of the highest intensity of each mass peak for previous scans and transfer of data from the PET to an LSI-11 system for data processing, plotting or printing. This system is now operational and is providing better results for depth profiles with the IMMA.

IV. Cumulative List of Publications and Papers Presented

1. C. J. Wu and D. B. Wittry, "Investigation of Minority Carrier Diffusion Lengths in GaAs', J. Appl. Phys., 49, 2827-2836 (1978).

2. D. B. Wittry, "Optimization of Recording Secondary Ion Mass Spectra", Proc. 13th Annual Conference of the Microbeam Analysis Society (1978).

3. D. B. Wittry and T. A. Whatley, "Energy Distribution of Positive Ions of Group III and Group V Elements Sputtered from Semiconductors", Proc. 13th Annual Conference of the Microbeam Analysis Society, 1978.

4. J. C. Potosky and D. B. Wittry, "The Secondary Ion Optics of a Quadrupole Ion Microprobe", Proc. 13th Annual Conference of the Microbeam Analysis Society, 1978.

5. F. Guo and D. B. Wittry, "Use of Specimen Current Integration in SIMS", Proc. 13th Annual Conference of the Microbeam Analysis Society, 1978.

6. D. B. Wittry, S. Y. Yin, and R. A. Wittry, "A Preliminary Evaluation of Ion Extraction Geometries Used in Secondary Ion Mass Spectrometry", Report on the Second Japan-U.S. Seminar on SIMS - Secondary Ion Mass Spectrometry: Fundamentals and Applications, Takarazuka, Japan, Oct. 23-27, 1978, pp. 211-213.

7. D. B. Wittry and F. Guo, "Adaption of the Dietz Detector to an ARL Ion Microprobe Mass Analyzer", Proc. 14th Annual Conference of the Microbeam Analysis Society, San Francisco Press, 1979, pp. 341-2.

8. F. Guo and D. B. Wittry, "Secondary Ion Mass Spectrometry Using Selected Primary Ions and Variation of the Partial Pressure of Selected Gases in the Specimen Region", Proc. 14th Annual Conference of the Microbeam Analysis Society, San Francisco Press, 1979, pp. 333-4.

9. D. B. Wittry and F. Guo, "Digital Mass Control for an Ion Microprobe Mass Analyzer", Proc. of the Second International Conference on Secondary Ion Mass Spectrometry, Springer Verlag (1979), pp. 199-201.

10. R. S. Lisiecki and D. B. Wittry, "An X-ray Spectrometer Data Acquisition System with a Microprocessor-Based Computer", Proc. 15th Annual Conf. of the Microbeam Analysis Society, San Francisco Press, 1980. pp. 95-96.

11. S. Y. Yin, "Eighteen Years of Secondary Ion Mass Spectrometry - A Bibliography of SIMS 1958-1975, Proc. 15th Annual Conf. of the Microbeam Analysis Society, San Francisco Press, 1980, pp. 289-311.

12. D. B. Wittry, "Spectroscopy in Microscopy and Microanalysis: The Search for an Ultimate Analytical Technique", Electron Microscopy 1980, Seventh European Congress on Electron Microscopy Foundation, pp. 14-21.

13. S. Y. Yin and D. B. Wittry, "Cathodoluminescence Study of the Thermal Conversion of GaAs", to be submitted for publication.

14. S. Y. Yin and D. B. Wittry, "Cathodoluminescence and SIMS Study of the Anodic Oxides on GaAs", to be submitted for publication.

15. D. B. Wittry and F. Guo, "A Simplified Hybrid Detector for Ion Counting", to be submitted for publication.

V. Appendix

Manuscripts of papers to be published.

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